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NASA TECHNICAL MEMORANDUM

NASA TM-82527

THE FEASIBILITY OF LOW-G GREY SOLIDIFICATION OF NODULAR IRON IN THE F-104 EXPERIMENTAL FURNACE PACKAGE

By P. A. Curreri, G. A. Smith, and G. Workman

(NASA-TM-82527) THE FEASIBILITY OF LOW-G GREY SOLIDIFICATION OF NODULAR IRON IN THE F-104 EXPERIMENTAL FURNACE PACKAGE (NASA) 20 p HC A02/MF A01 CSCL 22A N85-18996 Unclas G3/12 14331

April 1983

NASA

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama

TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO. NASA TM-82527	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.
4. TITLE AND SUBTITLE The Feasibility of Low-G Grey Solidification of Nodular Iron in the F-104 Experimental Furance Package		5. REPORT DATE April 1983
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) P. A. Curreri, G. A. Smith and G. Workman		8. PERFORMING ORGANIZATION REPORT #
9. PERFORMING ORGANIZATION NAME AND ADDRESS George C. Marshall Space Flight Center Marshall Space Flight Center, Alabama 35812		10. WORK UNIT NO.
		11. CONTRACT OR GRANT NO.
12. SPONSORING AGENCY NAME AND ADDRESS National Aeronautics and Space Administration Washington, D.C. 20546		13. TYPE OF REPORT & PERIOD COVERED Technical Memorandum
		14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

Prepared by Space Science Laboratory

16. ABSTRACT

The rationale for low-g experiments with cast iron and the need for solidification in the grey form during these experiments are reviewed. The factors which determine whether an iron melt will solidify grey or white are discussed. Cooling rate versus microstructure was studied for a nodular iron candidate material for F-104 low-g solidification. The study determined that low-g grey solidification, using the present F-104 furnace system, of the nodular iron composition studied is not feasible. Specimen microstructure strongly suggested that the F-104 furnace's gas cooling system was causing excessive localized chill resulting in the nucleation of the unwanted iron carbide phase. A change is suggested, in the quench system design, that could possibly overcome this problem.

17. KEY WORDS Low-Gravity Solidification, F-104, Nucleation, Growth, Grey Iron, White Iron, Critical Cooling Rate, Ductile Iron	18. DISTRIBUTION STATEMENT Unclassified - Unlimited P. A. Curreri

19. SECURITY CLASSIF. (of this report)	20. SECURITY CLASSIF. (of this page)	21. NO. OF PAGES	22. PRICE
Unclassified	Unclassified	19	NTIS

MSFC - Form 3292 (May 1969)

For sale by National Technical Information Service, Springfield, Virginia 22151

ACKNOWLEDGMENTS

We acknowledge the assistance of the personnel of John Deere and Company during this study especially that of Larry L. Fosbinder and John Mayberry in providing samples and compositional data.

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INTRODUCTION

A materials processing threshold report, completed February 1980 [1], suggested that low-g experiments with cast iron offer an excellent opportunity to study the effects of convection and sedimentation on alloy solidification. During the solidification of hypereutectic irons, density differentials between light graphite material floating in heavier liquid could be expected to cause gravity-driven segregation. Other density differentials in the melt could be caused by the segregation of lighter alloying components such as sulfur or of heavier alloying components such as rare-Earth elements. Thus, it was postulated that gravity levels during solidification would have a significant effect on growth, macrostructural heterogeneity, and the size and distribution of macrophases in cast iron.

The report recommended that samples solidified in low-g be compared to control samples solidified at 1-g to determine the role that gravity plays in terrestrial cast iron solidification. If it is determined that gravity has significant influence on cast iron solidified at 1-g, this knowledge might then be applied to commercial production of cast iron. A series of low-g scoping experiments were recommended to determine if detailed low-g study of cast iron solidification should be supported by the NASA Materials Processing in Space Program.

In June 1981, NASA began a Technical Exchange Agreement (TEA) with John Deere and Company [2] to collaborate in a series of low-g solidification experiments of commercial cast iron utilizing NASA's low-g aircraft [3,4]. The TEA agreement stated that particular attention, during this study, should be paid to finding the effects of low-g on graphite nucleation and growth.

The first series of experiments involved gas quenching of cast iron melts during the 20-sec, low-g time afforded by a KC-135 low-g parabola. In general, no difference between the low-g solidified samples and those solidified terrestrially could be ascertained from the results of these experiments. However, the analysis of some samples did suggest that nodular iron with more of the sample solidified in the high-g portion of the parabola tended to have more larger, perhaps aggregated, graphite nodules than samples solidified with a greater portion of their solidification in the low-g portion of the parabola [5]. Buoyancy-driven segregation and aggregation of graphite nodules has been observed in terrestrially solidified cast iron and studied by nodular iron solidification in high-g centrifuges [6]. The study of ductile iron solidification in the absence of nodule flotation might be of value. However, the KC-135 experiments were inconclusive because of poor reproducibility in the gas quench cooling rates and because complete solidification of the iron sample in the 20-sec, low-g period could not be attained without producing predominantly white iron.

In order to meet the TEA objective of emphasizing the study of gravitational effects on the growth and nucleation of the graphite phase of cast iron, it is essential to solidify samples predominantly in the grey form. Cast iron can, however, solidify as either of two eutectics, depending on the solidification conditions. The

first is the grey eutectic, of austenite and graphite, which is thermodynamically stable; the second is the white eutectic, of austenite and cementite (iron carbide), which is thermodynamically metastable. The reason that the metastable white iron can form is that although the grey eutectic nucleates more easily than the white [7], the white eutectic, once nucleated, has a faster growth rate than the grey [8]. This is the reason for the well-known phenomenon that a cast iron melt can be made to solidify grey by slow cooling or to solidify white by rapid cooling.

For a given sample-furnace configuration, critical cooling rates can usually be established for various iron compositions for grey or for white solidification [9]. However, the fundamental importance of nucleation and growth kinetics in determining whether white or grey iron will solidify has been demonstrated by hysteresis experiments [8,10]. These directional solidification experiements established that for pure Fe-C the grey to white transition can occur at rates at least 40 times faster than the white to grey transition.

It is also common for samples cooled at intermediate rates to solidify partially grey and partially white (mottled). Studies have shown [8] for such castings that normally the grey iron solidifies first, then at some point cementite is nucleated and rapidly grows to complete the solidification.

The irreproducibility of the cooling rates during the initial KC-135 iron solidification experiments being obviously not acceptable, a new furnace was constructed with an automatic meterable gas quench system. The furnace was built to be compatible with an F-104 aircraft that has the capability to extend the low-g period an additional 30 sec to a total of 50 [4]. Reproducible control of the furnace quench system makes it possible to conduct a systematic study of sample microstructure versus cooling rate for the sample compositions that are candidates for flight experiments. Thus, the main objective of this study was to determine the cooling rates necessary to solidify commercial nodular iron in the grey state using the F-104 experiemental furnace system and to design, if possible, a low-g solidification experiment for this material.

PROCEDURE

Commercial nodular iron was obtained as bar stock [11]. The chemical analysis of the sample obtained by mass spectroscopy and wet chemical analysis is given in Table 1. From this bar, individual samples were machined to fit cylindrical crucibles of high-purity alumina 12 mm in diameter and 25 mm in length. A cross sectional schematic of the crucible, which includes the thermocouple placement, is given in Figure 1. The construction and and operation of the F-104 furnace system has been described in detail elsewhere [12]. A schematic of the furnace canister, illustrating the gas quench system, is given in Figure 2. The cooling rate of the sample is set by adjustment of a needle valve and a pressure regulator, which control the helium gas flow to the quench gas inlet, until the desired cooling rate is obtained in a control sample. The variability in the cooling rate measured by the thermocouple shown in Figure 1, after the quench system has been set, is approximately $\pm 13°C/min$. The imbedded thermocouple, used to obtain the cooling data, was a type S of 0.062 in. in diameter placed in an alumina thermocouple insulator and covered with an Inconel sheath. After each sample was solidified, it was sectioned longitudinally and polished and etched with 2 percent Nital as preparation for photometallography.

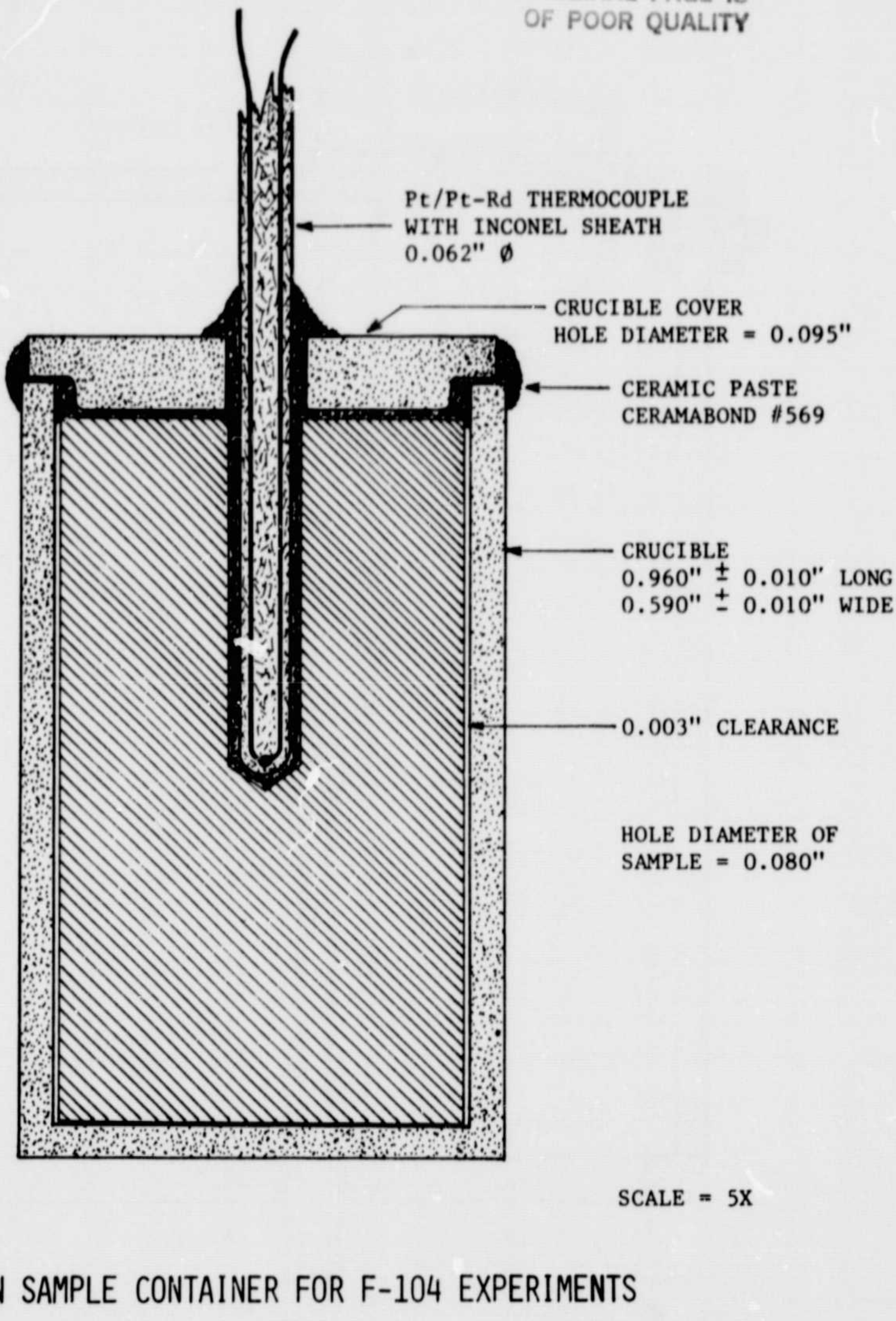

Figure 1. Cross Sectional Schematic of the F-104 Experimental Furnace Crucible Showing the Thermocouple Arrangement.

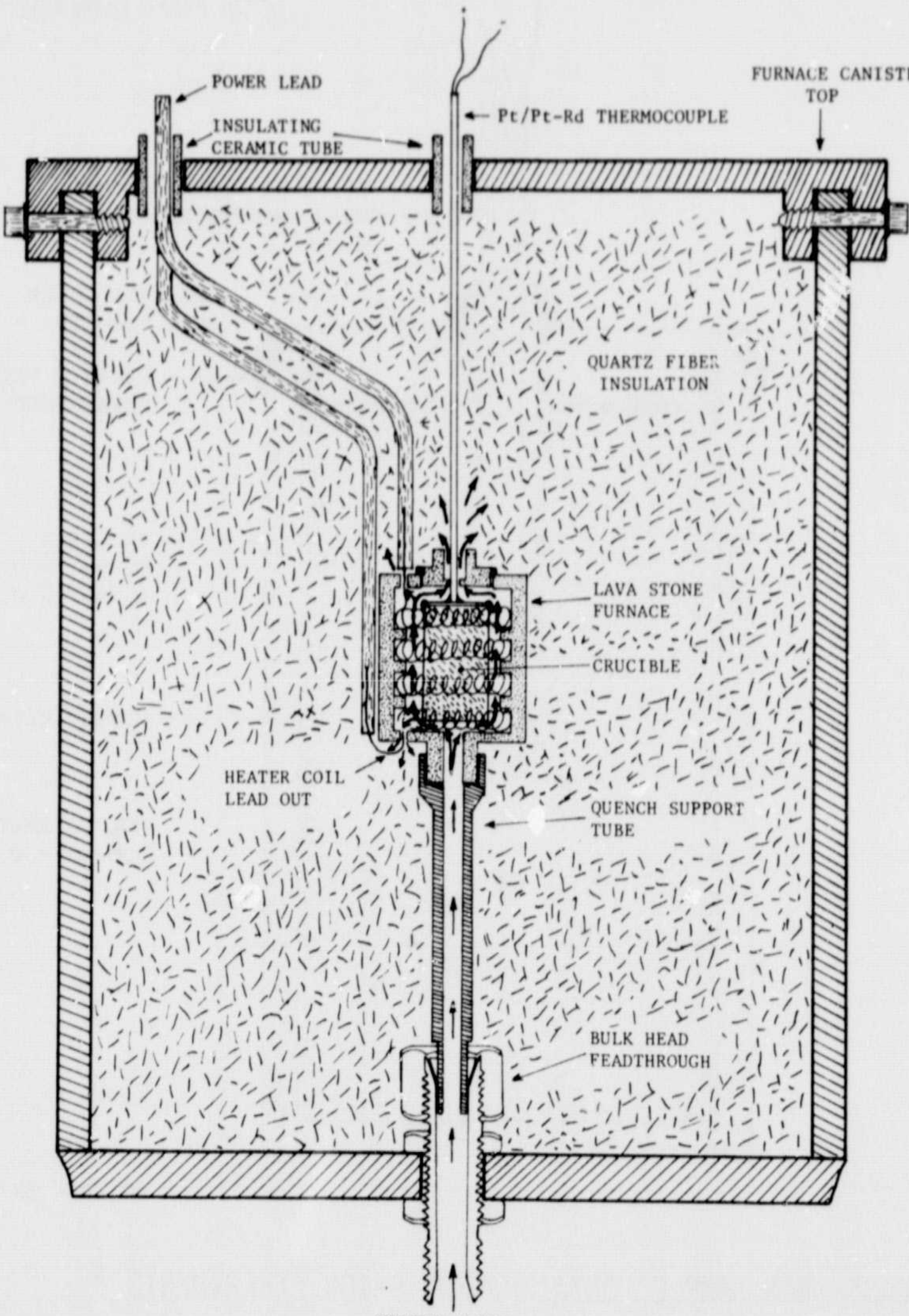

Figure 2. Cross Sectional Schematic of the Furnace Compartment. The Arrows Designate the Quench Gas Flow.

TABLE 1. CHEMICAL ANALYSIS OF NODULAR IRON SAMPLES

Element	C	S	Si	Mg	Cu
Wt%	3.73	0.005	2.79	0.055	0.26

Element	Mn	Cr	Ce
Wt%	0.45	0.052	0.013

RESULTS

Figures 3 through 7 show the thermal histories and microstructures of samples solidified at five different gas quench settings. The cooling rates, R1, listed in the figures were determined by graphically measuring the slope of the cooling curves just prior to the thermal arrest at solidification.

Figure 3 shows the sample solidified at the lowest cooling rate, 76°C/min. This rate was that obtained by natural cooling of the furnace - without the use of the gas quench. From the micrographs it is apparent that this sample solidified in the grey state. Numerous graphite nodules are visible surrounded by bull's-eye ferrite. The deterioration in shape of many of the nodules indicates that the holding time before solidification was long enough to decrease the effectiveness of the magnesium inoculation.

The samples shown in Figures 4 through 7 were solidified with the use of the helium gas quench system.

Figures 4 through 6 show samples solidified at cooling rates of 188°, 215°, and 261°C/min, respectively; all have a mottled microstructure. Numerous graphite nodules and massive carbide platelets (white) are present simultaneously. The amount and size of the carbide platelets increased as the cooling rate of the sample increased. Examination of these samples revealed a chill zone of white iron on the surface of the sample that faced the helium quench gas inlet. It was also observed that all the carbide platelets, except those at the crucible walls, are predominantly oriented parallel to the longitudinal axis of the crucible.

Figure 7 shows a sample that was cooled at a rate of 285°C/min before solidification. This sample solidified completely as white iron. There are no apparent graphite nodules in the sample, and a dendritic pattern of austenite (black) can be seen alongside massive carbide (white).

DISCUSSION

Figure 8 shows a schematic representation and accelerometer recording of the g-levels versus time in a typical F-104, low-g maneuver. Table 2 summarizes the results shown in Figures 3 through 7. R2 is 150°C divided by the time it took the sample to cool from 1250° to 1100°C, and TA is an estimate of the amount of time the sample spent at the solidification thermal arrest. An objective defined during the planning of the cast iron aircraft experiments has been to solidify the iron in the grey state while cooling it from 1250° to 1100°C during the 20 or 40 sec of good low-g. This, it was felt, would assure that the sample had completely solidified in

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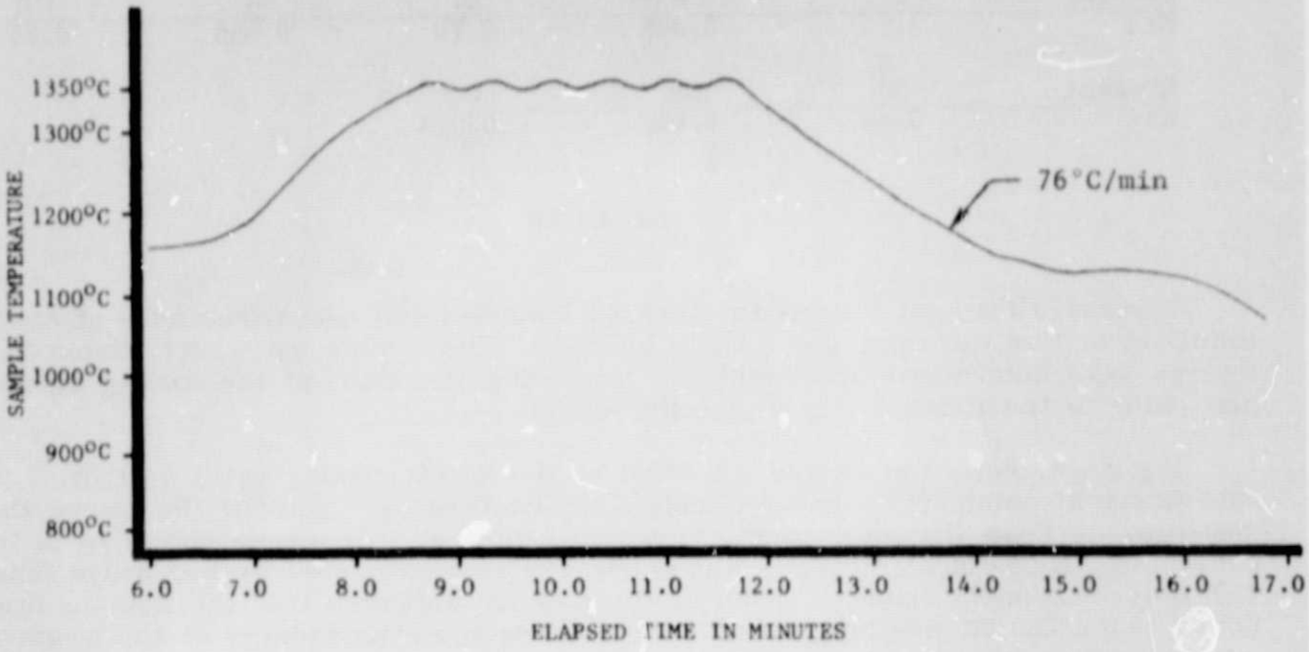

Figure 3. Thermal History and Photomicrographs for Nodular Iron Cooled at 76°C/min.

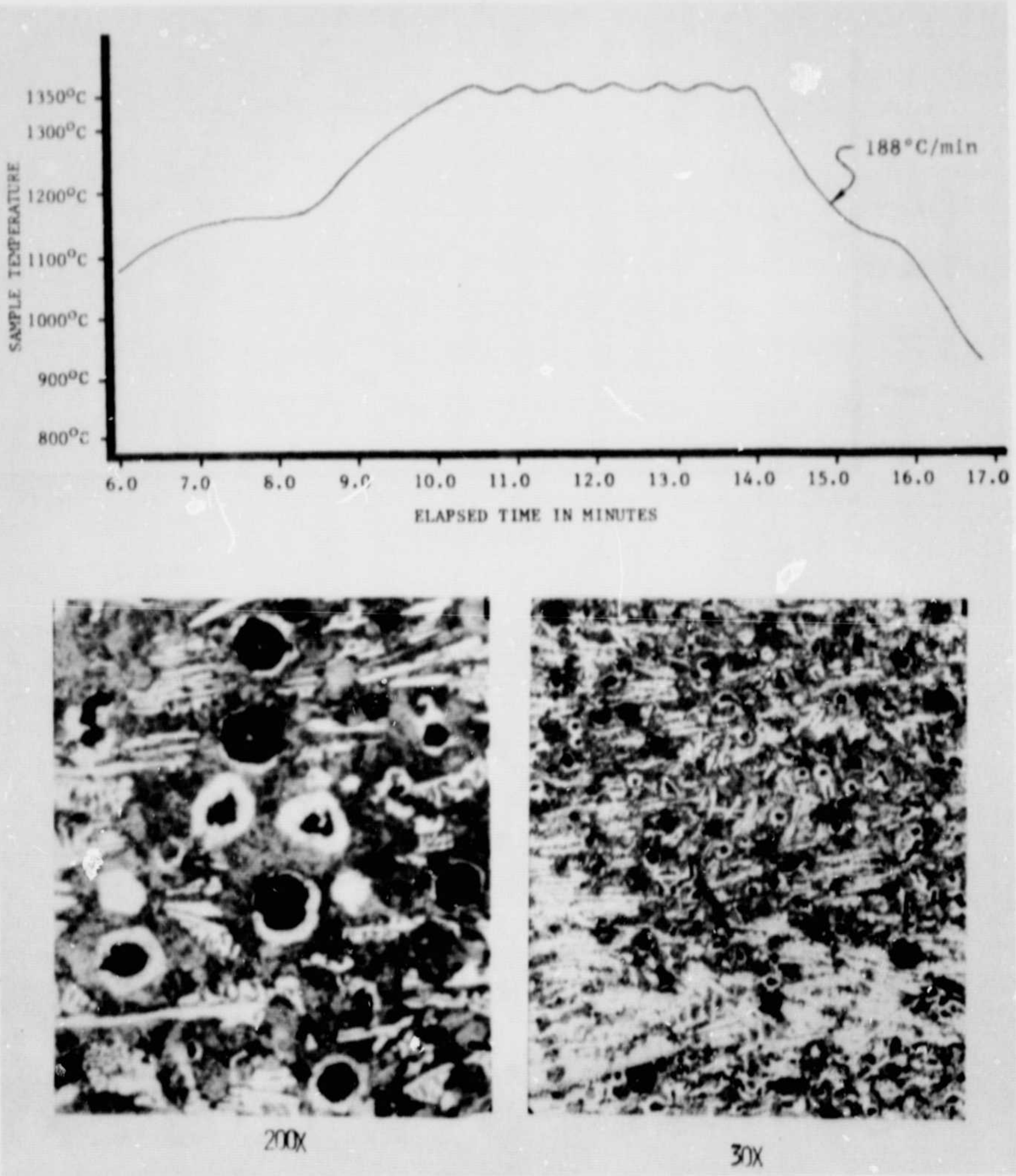

Figure 4. Thermal History and Photomicrographs for Nodular Iron Cooled at 188°C/min.

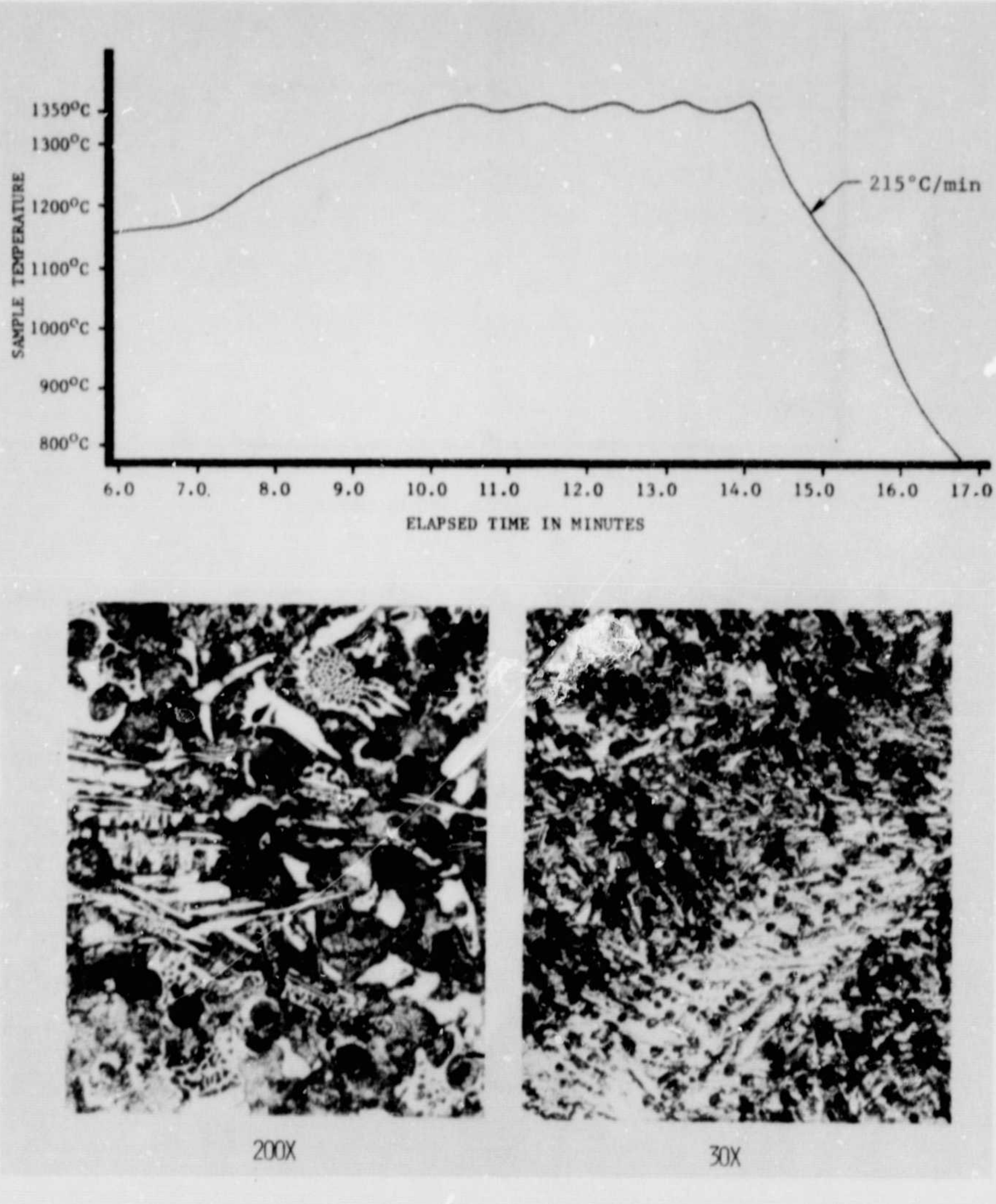

Figure 5. Thermal History and Photomicrographs for Nodular Iron Cooled at 215°C/min.

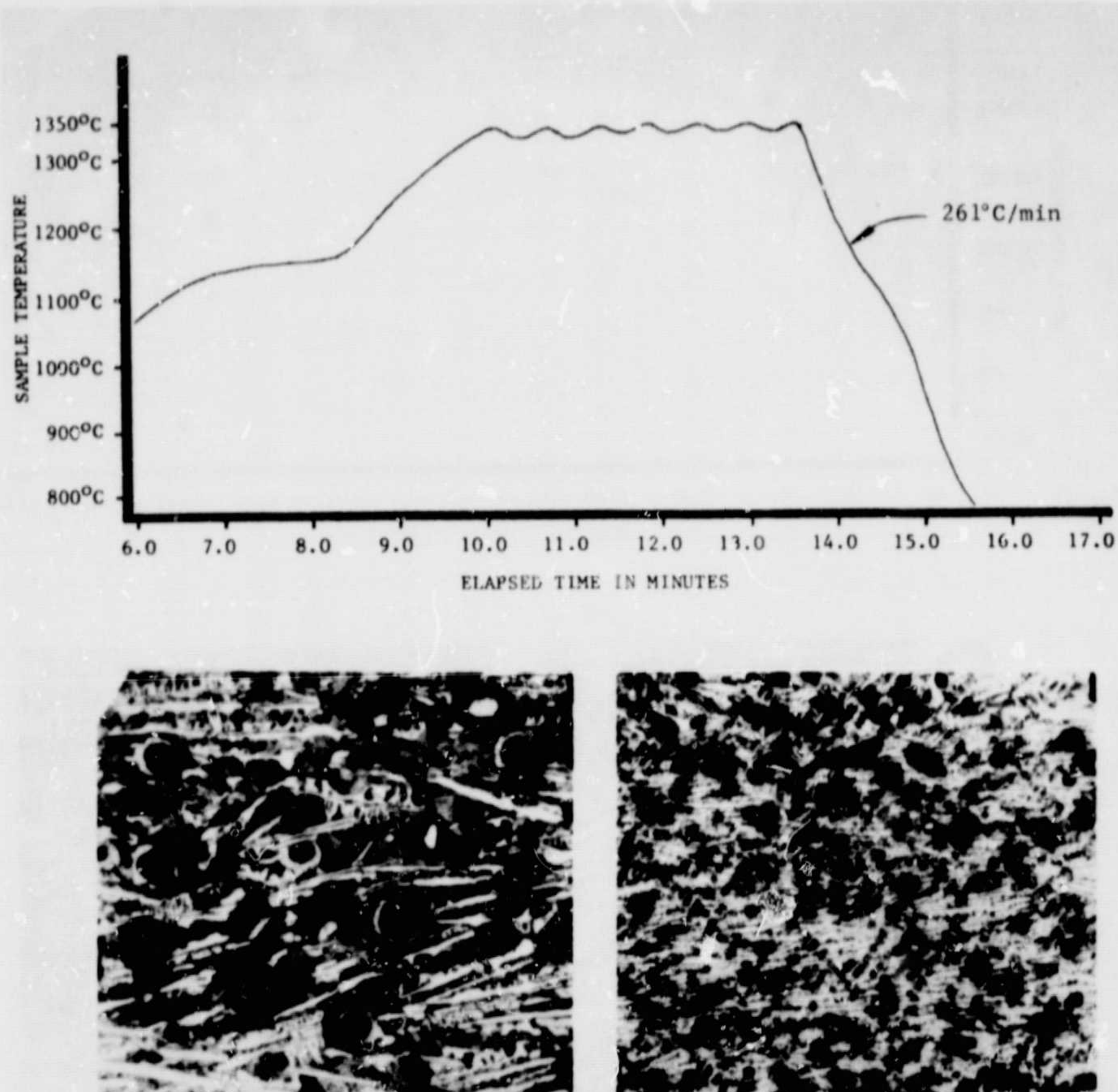

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Figure 6. Thermal History and Photomicrographs for Nodular Iron Cooled at 261°C/min.

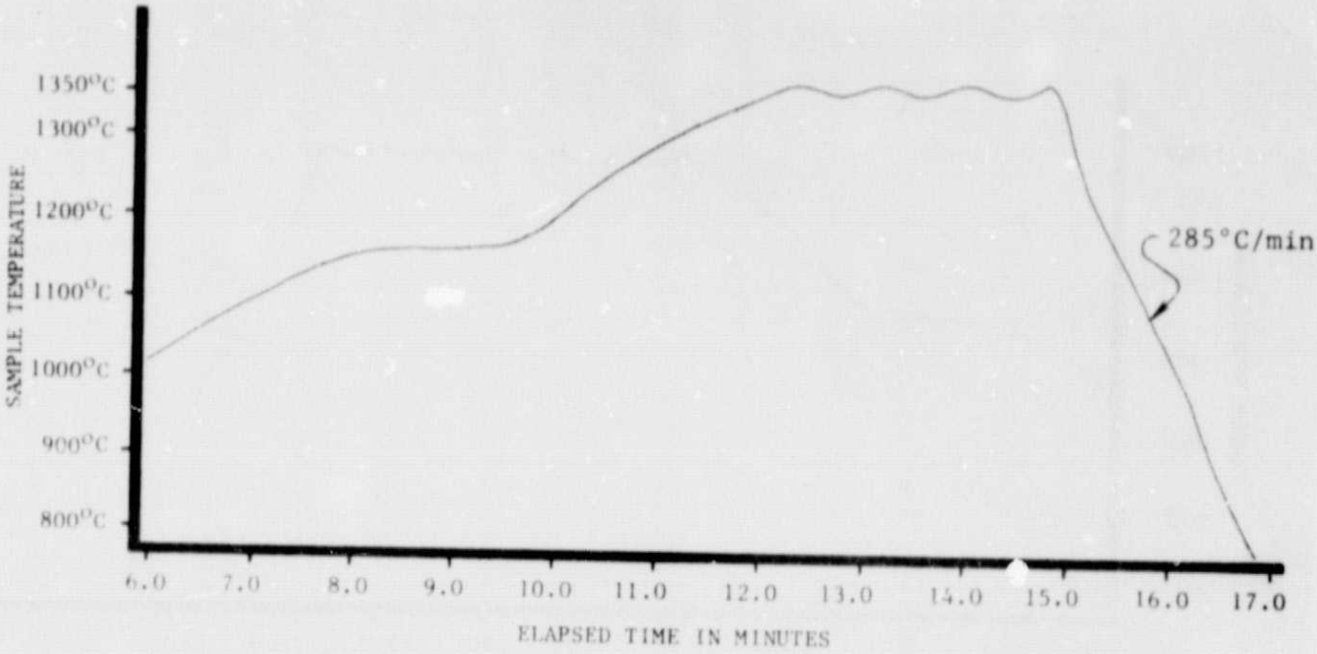

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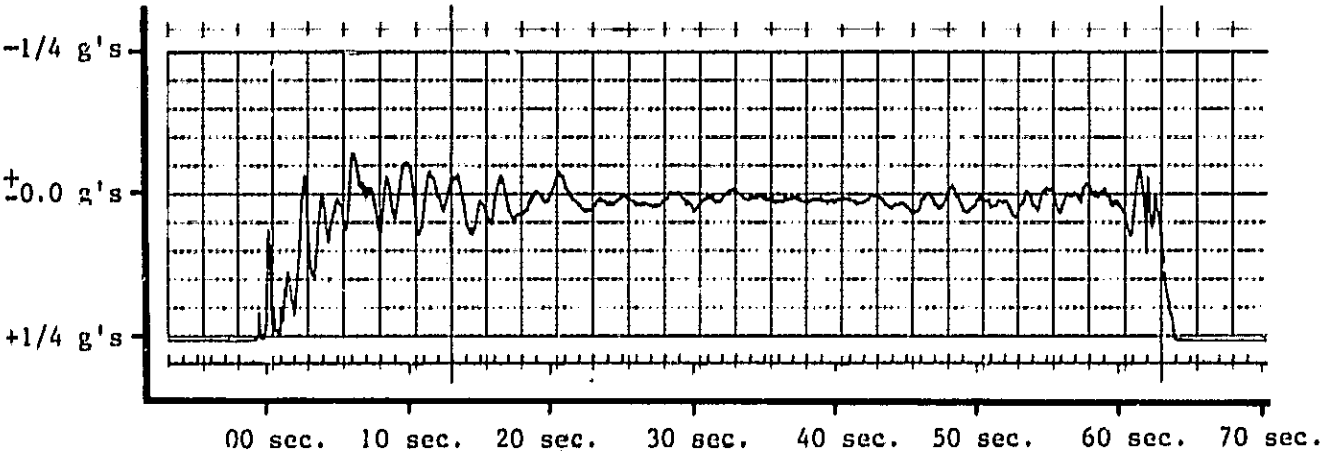
Figure 7. Thermal History and Photomicrographs for Nodular Iron Cooled at 285°C/min.



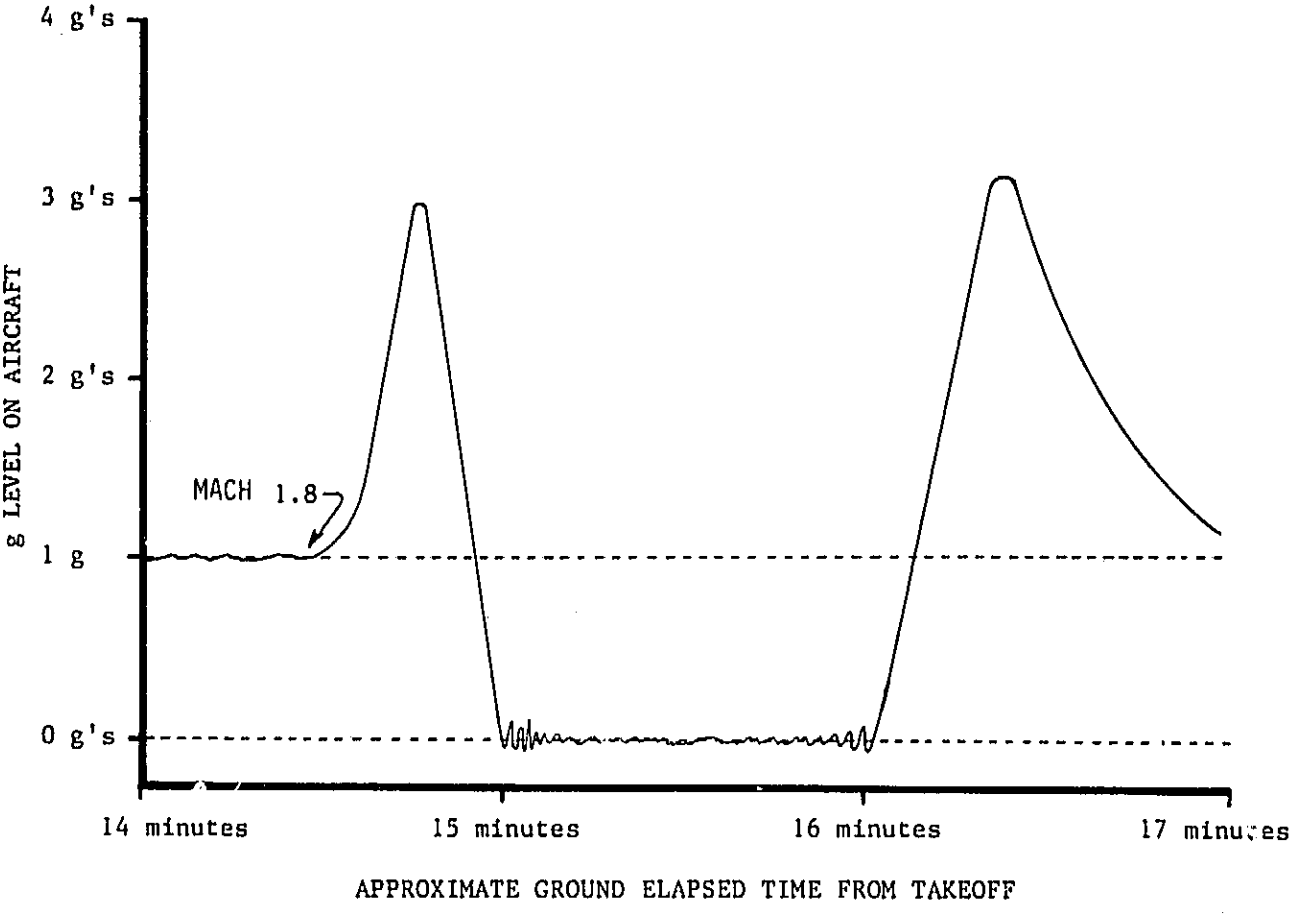


Figure 8. Accelerometer Data and Schematic Showing Gravity Levels and Their Duration for a Typical F-104, "Low-g" Maneuver.

TABLE 2. SUMMARY OF COOLING RATE AND MICROSTRUCTUAL DATA

R2 (°C/min)	R1 (°C/min)	TA (min)	Microstructure
43	76	2.6	Grey
109	188	1.3	Mottled
187	215	0.8	Mottled
223	261	0.6	Mottled
267	285	0.5	White

low-g. It can be seen in Table 2 that the rate, R2, for grey solidification would be somewhere between 42 and 109°C/min. It is obviously not possible to achieve this objective with the present sample-furnace configuration. Marginally, it could be argued that most of the sample solidified in low-g if at least the thermal arrest fully occurred in low-g. From the estimate of the thermal arrest time in Table 2, it can be seen that even with precise experimental sequencing this minimal criterion would not likely be achievable.

The microstructural examination of the samples in this study also revealed strong evidence of excessive localized quench. This is apparent from the white iron chill zone in the mottled samples at the surface facing the quench gas inlet (Fig. 2). Further evidence for excessive localized quench is the observation that all the carbide platelets, except those very close to the crucible surface, orientated themselves with their edges facing the direction of the gas quench inlet. Studies have shown [8] that when a steep gradient is applied to a small casting during solidification, the cementite orientates itself in such a way that its edgewise growth will take place in the direction of the thermal gradient (since the edgewise growth of cementite tends to be most rapid). Finally, there is the fact that the only sample that solidified completely grey in this study was the sample in which no gas quench was used. All of these observations very strongly indicate that the gas quench impinging on the bottom crucible surface is causing the nucleation of iron carbide and thus promoting the solidification of mottled iron.

Redesign of the gas quench system so that the heat is extracted evenly from the sample's surface could result in grey iron microstructure at the same effective cooling rates - as measured by the thermocouple in the sample center - that now yield mottled iron. Samples of this composition can be cooled at rates as high as 300°C/min, under different solidification conditions, and still yield grey microstructure [11]. Thus, grey solidification of nodular iron in low-g using the F-104 experimental furnace package should become feasible with the optimization of the gas cooling system.

CONCLUSIONS

1) Low-g solidification of grey nodular iron is not feasible with the current F-104 experimental furnace system for the iron composition studied.

2) The current gas quench system produces excessive localized chill that has the effect of nucleating the unwanted iron carbide phase. Since the iron carbide

phase grows at a much faster rate than the grey phase, this results in mottled or white microstructures.

3) The redesign of the F-104 furnace quench system to extract heat more evenly from the sample's surface could produce grey castings at cooling rates that are now producing mottled castings.

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APPROVAL

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

A. J. Dessler

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☆ U.S. GOVERNMENT PRINTING OFFICE 1983—646-058/195